THE ORIGIN OF SPIRAL GROWTH DEFECTS DURING EARLY STAGES OF HOMOEPITAXIAL DIAMOND GROWTH ON (100) SURFACES

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Abstract

Interrupted growth experiments and topographical characterization using atomic force microscopy and scanning electron microscopy were used to understand the origin of defect formation during homo-epitaxial growth on well polished, diamond {100} substrates. Experimental evidence suggests that the origin of spiral growth patterns and trapezoidal growth hillocks could be traced to "mis-aligned" joining of two islands during growth on {100} substrates. The growth of mis-oriented islands on the well polished, diamond {100} surfaces occurred due to the local mis-tilts of the lattice caused by polishing in <110> direction. The results also showed that when the sub-surface damage due to polishing was removed, the defect formation was minimized and a layer by layer growth mode was observed. Further experiments are needed to further understand the reasons for observed spiral growth patterns under cases where no stresses are present, i.e., spiral growth patterns on the {100} facets of vapor phase grown cubo-octahedral shaped crystals.

Introduction

Understanding the formation and propagation of defects at the atomistic scale is the key to the development of processes for synthesis of large area, defect free diamond. Homoepitaxial growth on {100} substrates is typically used for obtaining films with smoother surfaces compared to (111) and (110) surfaces. However, homoepi growth on (100) surfaces frequently results in several characteristic defects such as: pyramidal growth hillocks, trapezoidal features and spiral growth patterns. Although, the nature of these defects is well known in terms of their geometric character, the origin of such defects is poorly understood. Several variations in growth conditions have been attempted to eliminate these defects and to obtain defect free films. These methods primarily relied on varying the growth temperature and feed gas compositions (ultra-low CH_4 concentrations) which typically result in low growth rates. Many of the studies have used topographical characterization after final stages of growth on thicker films to observe the defect formation, which cannot be used to determine the origin of defects. A better understanding of the origin of defect formation during early stages would allow one to develop "high growth rate" recipes for homoepi of diamond.

Here, we conducted a set of interrupted growth experiments followed by topographical characterization using AFM and SEM. The goal of these studies was to capture the early stages of defect formation and to follow the evolution of the same defect over a sequence of several growth runs. Such a morphological evolution sequence of defects, with respect to its local environment, is used to track the early stages of defect formation (or spiral growth pattern). However, due to the lack of large enough atomically smooth surfaces, mechanically polished (100) surfaces of HPHT type Ib diamond substrates were used.

Experimental

Growth experiments were performed in a MW plasma reactor using 0.33% CH_4/H_2 feed gases, 50 torr reactor pressure and 1100 watts microwave power. Same growth conditions were maintained throughout all the experiments. The as-received diamond substrates were mechanically polished and acid cleaned before any growth was performed on them. The mis-orientation of all the (100) surfaces used here, was maintained less than 1^o. In order to capture the early stages of defect formation, growth experiments were performed for duration of a few minutes, followed by topography analysis using AFM/SEM and optical microscopy. Several such sequential growth experiments were performed on the same substrates to obtain the morphological evolution of defects. Two different sets of substrates were used in these studies: (a) (100) substrates with <110> mechanical polishing grooves and (b) specially prepared (100) surface by removing the sub-surface damage caused by mechanical polishing.

Results

Experiments performed on substrates that were mechanically polished in the [110] direction resulted in typical defects on the surface. These defects occur primarily along the polishing grooves. Interrupted growth experiments performed in this work allowed us to determine the primary reason for defect formation. An example of the formation of a spiral defect is shown in Figure 1. The formation of the secondary crystallites, typically observed on defects on (100) surfaces, is identified to be due to the nucleation of a twined crystal on a local {111} plane appearing during the mis-aligned joining of growth islands. Homoepi growth on substrates that were treated for removing the mechanical polishing induced sub-surface damage resulted in defect free layers.

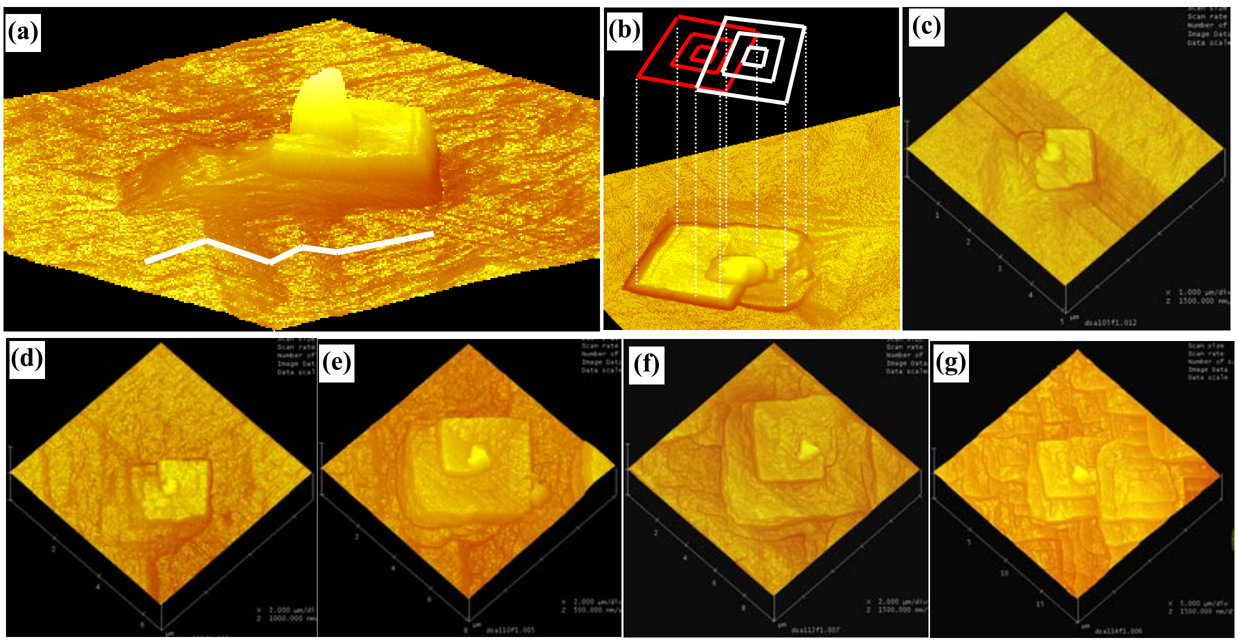

Figure 1. Formation of a spiral defect. (a) a perspective view of the defect during the early stages of growth indicating the role of polishing grooves (highlighted by white line), (b) different perspective view of the defect shown in (a) with increased z-scale and indicating the mis-aligned joining of two islands (shown in red and white and (c-g) morphological evolution sequence of the same defect shown in (a) and (b) over a sequence of 5 growth experiments, each lasting 1 hr.

The typical evolution sequence of a spiral growth pattern as shown in Figure 1 strongly suggests that the island formation on either side of the groove is favored and their joining during growth typically resulted in a mis-aligned joining as shown in Fig. 1(b). Further growth after joining led to spiraling of uneven surfaces. Earlier studies (1, 2) have revealed that mechanical polishing could generate enough stresses to cause local mis-tilts on the top surface of diamond stones. These mis-tilts in regions around polishing grooves allowed for such mis-aligned joining of islands across the grooves, causing spiral growth patterns.

References

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